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Effects of Temperature and Cationic Surfactant on the Clarification of Sugar Syrup by Air Dissolved Flotation

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***Abstract*— *Impurities responsible for color and turbidity of dissolved crude crystal sugar in water can be separated by dissolved air flotation (DAF) process according to a specified refined sugar. Clarifying agents help to adsorb and precipitate these impurities forming precipitates, which adhere to micro bubbles generated by injected pressurized air, forming agglomerates that float and are removed from the surface. Experiments of DAF were conducted using cationic surfactant (150, 225 and 300 ppm), to clarify sugar syrup at 66°Brix, at temperatures of 26, 40 and 55°C, 895 kPa pressure, 300 s agitation, followed by 2 hours of phase separation. The clarification of sugar syrup was judged by physicochemical analyzes of conductivity, color, turbidity and filterability. The best result was obtained with 225 ppm clarifying agent and 40°C resulting 36% color and 98% turbidity removal, a 50% increase in the rate of filterability, although with 32% increase in ash content. The study has demonstrated that color and turbidity can be removed and filterability rate can be increased for the clarified sugar syrup by DAF process using only a cationic surfactant without adding any other chemical agent, and conducting the process at a relatively low temperature.*

***Keywords*— Clarify sugar syrup, Micro bubbles, sugar cane, sugar cane refinery, sugar syrup.**

I. INTRODUCTION

The process of dissolved air flotation (DAF) starts by generating and introducing air under pressure into the liquid medium, followed by sudden reduction in pressure and the generation of micro bubbles. Air is injected into the liquid through a saturator under pressure, and after saturation, the mixture is injected in the flocculation tank at atmospheric pressure, where the excess of dissolved air is released in the form of micro bubbles, which in turn adhere to the solid phase precipitated in the liquid medium, forming agglomerates of particles and micro bubbles. These agglomerates float to the surface due to their lower density, from which they are easily removed as foam (CREMA, 2012; CREMA-CRUZ, 2017).

The main task of a sugar cane refinery is to remove the color and reduce the amount of impurities (non-sugars) present in the crude sugar crystals, used as starting material, in order to produce a commercial sugar with a higher degree of purity. For the refining process, crude crystal sugar is dissolved in water, resulting in a dense solution, called "sugar syrup" or sometimes “melt liquor”, with the purpose of diluting the residual film (mother liquor) surrounding the crystals, facilitating the clarification by flotation and subsequent process steps to the production of refined sugar (RIBEIRO, 2003; REIN, 2013).

Among the various unit operations involved in the production of refined sugar, the clarification of the sugar syrup is of major importance, with great influence in the manufacture of sugar, not only on the quality and product application performance, but also on subsequent production steps (REIN, 2013). The objective of the process is to obtain a limpid, clear syrup by coagulation, floculation and subsequent removal of impurities (non sugar) suspended and dispersed, responsible for the increased color and turbidity, and products with low purity and quality (JENKINS, 1966, REIN, 2007).

The coagulation is a process consisting of two subsequent phases. In the first stage coagulation occurs by adding chemical coagulants to reduce the repulsive

forces between particles in suspension. In the second phase, flocculation occurs due to collisions between destabilized particles by coagulation, forming larger particles (RICHTER, 2009).

The principle for color removal from sugar syrup by refineries is based on cationic charged added molecules that interact more strongly with the anionic organic impurities, present in the syrup, than the added Ca^{2+} in the liming process (DOHERTY, EDYE, 1999). For this purpose, it is used cationic surfactants (mainly containing active polyamine), known as color precipitating (or decolorizing) because positive charges combine with the colored compounds, generally negatively charged at processing pH (COPERSUCAR, [199-?]; REIN, 2013). Moreover, in almost all flotation processes, selective hydrophobicity of the particles is achieved by adsorption of surfactants to make the hydrophilic surfaces of particles into hydrophobic surfaces, which are easily attached to the inserted air micro bubbles, by presenting a net negative charge due to adsorption of negative ions (HOLMBERG et al., 2002).

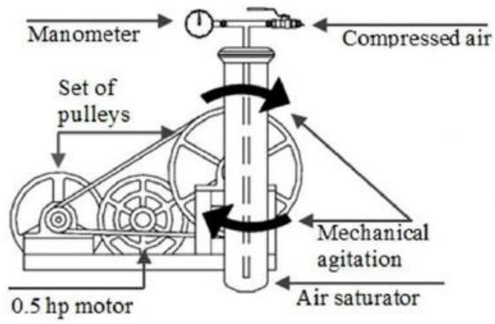
Cationic surfactants have been developed (Talofloc®, Cyanamid®, Talomel®, etc.), with the purpose of precipitating color compounds in the syrup. The mechanisms for the precipitation are quite similar, but with the different and restrictive dosages according to the Food and Drug Administration (FDA) (CHOU, 2000; REIN, 2013). Other methods (liming, phosphatation, carbonation, etc.) are widely applied to assist the removal of non sugar compounds present in sugar syrup. Cationic surfactants promote changes in surface charges of the compounds of non-sugars, promoting coagulation and flocculation. Furthermore, the clarification process is typically conducted at high temperature (nearly 82°C), however, the exposure of the syrup to high temperature for a certain time and low pH may result in loss due to sucrose inversion.

This study aimed to evaluate the influence of operating temperature and dosage of cationic surfactant for clarifying sugar syrup at 66°Brix (g soluble solids/100 g) by dissolved air flotation (DAF) process.

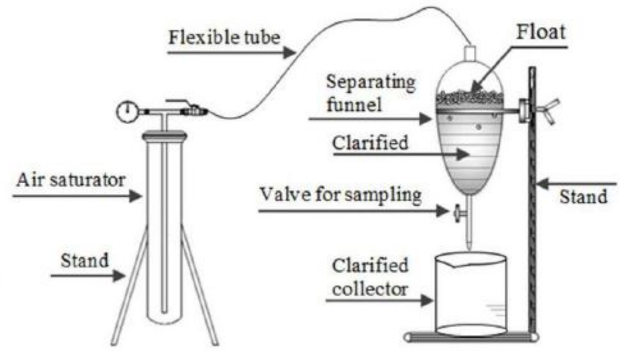
II. MATERIAL AND METHODS

2.1. Flotation Apparatus (Air Saturator)

The DAF experiments were conducted in an air saturator bench scale apparatus constructed of stainless steel, consisting of 3 in diameter and 0.45 m height a vertical cylinder, with a total volume of 2 L, capable of treating 1 L of liquid per batch. In this cylinder is attached a cover and a vertical ½ in diameter filtered pressed ar stainless steel tube for on pressed air injection (Fig. 1).



(a)



(b)

Fig. 1: Scheme of the flotation apparatus: (a) air saturator coupled to mechanical agitator, with the movement indicating arrow; (b) flotation system coupled to the phase separation setup.
Source: Authors.

The system has a sealing lid with a pressure gauge and a ¼" valve for the connection with compressed air and subsequent removal of liquid saturated with air after pressurization. The whole system is driven by a 0.5 hp motor, providing about 100 rpm translational agitation (Fig. 1a). For phase separation, a pear-type separation funnel was used, made of Pyrex® glass, 1000 mL capacity and a bottom valve, which allows easy removal of aliquots for collecting the clarified product (Fig. 1b).

2.2. Crude Crystal Sugar

Samples of crude crystal sugar cane from 2010/2011 harvest, was provided by Guarani Sugar Mill plant (Cruz Alta unit), located in Olympia, state of São Paulo. This crystal sugar is called by the industry as a feedstock for

the refinery, with 99.7% polarization, produced from cane juice.

2.3. Cationic Surfactant

The cationic surfactant Flonex SI 7080 (SNF Floerger, Skills Chemical, Dois Córregos, SP, Brazil) was used for neutralization and coagulation of particles (soluble, insoluble and colloidal) negatively charged present in the sugar syrup, consisting of an aqueous solution of the cationic surfactant of low molecular weight and high viscosity, containing polyamine as the active substance.

2.4. Preparation Of The Sugar Syrup

For the preparation, crude crystal sugar was dissolved in deionized water, pre-heated at 40°C, in a thermostat bath (Marconi MA-184, Piracicaba, SP, Brazil), under agitation of 1700 rpm by a mechanical stirrer (Quimis Q235, Diadema, SP, Brazil), until all sugar present was completely dissolved, forming a viscous and homogeneous syrup, with a concentration of 66 ±1°Brix and pH 5.8 ±0.2. After complete dissolution of the sugar, the temperature of the syrup was adjusted to the temperature of the experiment, and the sugar concentration adjusted with a digital refractometer (Atago PAL-3, Ribeirão Preto, SP, Brazil). The pH and temperature were measured directly by a digital pH meter and digital thermometer respectively (Tecnal TEC-11, Piracicaba, SP, Brazil).

The physicochemical analyzes were performed for each sugar syrup, prepared at the time of the experiments for clarification by DAF, to preserve its physical and chemical characteristics. The physicochemical characteristics of the sugar syrup are presented on Table 1, as average of the results of all solutions prepared for the experiments, based on the following parameters: soluble solids content (°Brix), color ICUMSA (International Commission for Uniform Methods of Sugar Analysis), turbidity NTU (Nephelometric turbidity units), conductivity and filterability.

Table 1: Physicochemical characteristics of the sugar syrup at 66°Brix.

Parameter	Value
Soluble solids content [°Brix]	66 ±1
Color ICUMSA [UI]	428 ±20
Turbidity [NTU]	5,00 ±1,00
Conductivity [%]	$7{,}13\text{x}10^{-3}$ $\pm 0{,}01\text{x}10^{-3}$
Filterability [(mL) (min^{-1})]	5,5 ±0,5

Source: Authors.

2.5. Air Dissolved Flotation (DAF) Experiments

For the clarification of sugar syrup (66 °Brix) by the DAF process, with cationic surfactant, the experiments were conducted based on a experimental design, using a 2^3 factorial design, for two factors (temperature and dosage of cationic surfactant) with three levels each (Table 2).

Table 2: Factorial design for the DAF experiments.

Experiment	Temperature (°C)	Surfactant (ppm)	Randomized order
1		150	6
2	26	225	9
3		300	2
4		150	7
5	40	225	4
6		300	1
7		150	8
8	55	225	3
9		300	5

Source: Authors.

The controlled variables were temperature (26, 40 and 55°C) and the dosage of cationic surfactant (150, 225 and 300 ±5 ppm). The DAF experiments were randomized to minimize experimental errors. The response variable was the clarification of sugar syrup, comparing the results obtained by physicochemical analyzes of the untreated syrup with the syrup clarified by DAF process. The operating temperature was kept constant by the use of an ultra-thermostatic bath (Marconi MA-184, Piracicaba, SP, Brazil) at the temperature of each experiment until the time of transfer of the treated syrup to the flotation apparatus.

To perform the DAF experiments, the syrup dosed with the cationic surfactant was gently stirred by a glass rod, transferred to the flotation apparatus, sealed, and subjected to the process at 895 kPa pressure. After reaching the desired pressure, the flotation apparatus is coupled to a mechanical agitator for 300 s, promoting turbulence and homogenization of the sample with injected air. Then the flotation apparatus is placed in a holder, the valve opened slowly for depressurization and transferring the biphasic mixture (syrup + micro bubbles of air) to a separating funnel, followed by the process of phase separation (clarified solution and floating agglomerates) for 2 hours. After completion of phase separation, aliquots of the clarified sugar syrup were

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collected from the bottom of the separating funnel, for the evaluation of the physical and chemical parameters.

The operating pressure of 895 kPa and time of 300 s for mechanical agitation were determined in a previous work (CREMA, DARROS-BARBOSA, 2011), conditions in which the concentration of dissolved air is maximum. It was observed that the earliest settling of floated particles occurs two hours after the beginning of the phase separation process.

The operating temperatures (26 to 55°C) and dosage range of the cationic surfactant (150 to 300 ppm) were determined in previous studies, in which it was demonstrated that it was possible for color removal for the clarification of sugar syrup by DAF process, using only the cationic surfactant, without adding any other chemical agent. The study also showed that there is loss of efficiency of the DAF conducted at higher temperatures (> 80ºC), as is currently used in Brazilian sugarcane mills and refineries, thus, recommending flotation at temperatures below 60°C. On the other hand, inadequate temperature (too low) may negatively influence the dispersion of the fining agents used during the clarification process, delaying coagulation and precipitation of particles responsible for the color and turbidity development in the syrup, and consequently becoming dificult the separation from the liquid medium.

2.6. Analytical Methods

The analytical methodology used for assessing the DAF for clarification of sugar syrup was based on the methods recommended by ICUMSA (2007) and Copersucar (2005, 2009, 2011), by sugar industries currently used. The parameters analyzed were conductivity ash (% by weight), ICUMSA color (UI), turbidity (NTU) and filterability [(mL) (min^{-1})].

The method CTC-MT1-LA-006, version 02/2009, using a digital conductivity meter (Analion Q279P, Ribeirão Preto, SP, Brazil), was used for the analyzes of conductivity ash. The color was determined by the ICUMSA method CTC-MT1-LA-007, version 05/2011 (GS2 / 3-9 (2005), using a digital spectrophotometer (Micronal B342II, São Paulo, Brazil) by the absorbance at wavelength 420 nm. For the analyzes of turbidity, the method CTC-MT1-LA-014 was employed, version 05/2011, using a digital turbidimeter (Quimis Q279P, Diadema, São Paulo, Brazil). The filterability was evaluated using MT1-CTC-LA-016 method, version 02/2009, by a vacuum filtration assembly.

The efficiency of flotation (ε (%)) for the physicochemical parameters of conductivity ash, color and turbidity was determined as a function of temperature and dosage of cationic surfactant, relating the initial value (untreated syrup) to the measurement of the clarified sugar syrup, by Equation (1).

$$\varepsilon(\%) = \left(\frac{C_{treated}}{C_{untreated}} - 1 \right) 100 \qquad (1)$$

where ε (%) is the flotation efficiency, related to the physicochemical parameter analyzed; $C_{untreated}$ is the value of the physicochemical parameter for the untreated syrup, calculated for conductivity ash (% by weight), ICUMSA color (UI), and turbidity (NTU); $C_{treated}$ is the value of the physicochemical parameter for the clarified sugar syrup by the DAF.

For the evaluation of the results of filterability, it should be observed that the higher the rate of filterability ((ml) (min^{-1})), better the clarification of the sugar syrup by DAF, contrary to the other parameters, which in turn they are expected to be reduced after clarification. Thus, Equation (2) was used to calculate the efficiency (%) for the parameter filterability.

$$\varepsilon(\%) = \left(\frac{C_{treated}}{C_{untreated}} \right) 100 \qquad (2)$$

where $C_{untreated}$ and $C_{treated}$ are, respectively, the initial value of the rate of filterability ((mL) (min^{-1})) for the untreated syrup and that of the clarified-treated sugar syrup by DAF.

2.7. Statistical Analysis

Assays were performed in triplicate and the results were analyzed for two factors (temperature and dosage of cationic surfactant) and three levels by the analysis of variance (ANOVA), through F-test and the averages compared by Tukey test at 95% probability, using the *software* statistic *Minitab 15* (Minitab Inc., State College PA).

III. RESULTS AND DISCUSSION

Tables 3 and 4 show the flotation efficiency (%) as well as the corresponding statistical results of the respective for the physicochemical parameters of the sugar syrup clarified by DAF as a function of

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temperature and dosage of the cationic surfactant, calculated by Equations (1) and (2). Fig. 2, 3, 4 and 5 show the results obtained, respectively, for conductivity ash, ICUMSA color, turbidity and filterability, as a function of temperature and dosage of the cationic surfactant the clarified sugar syrup obtained by the DAF process. For comparison, these graphs shows the value for the untreated sugar syrup (0 ppm of cationic surfactant).

Table 3: Results of the statistical analysis for physical and chemical parameters of the clarified sugar syrup by DAF as a function of temperature and dosage of cationic surfactant.

	Conductivity ash	**Color ICUMSA**	**Turbidity**	**Filterability**
Variable	**p-value**			
Dosage of cationic surfactante	< 0,000*	0.123	< 0.000*	< 0.000*
Temperature	0,009*	0.170	< 0.000*	0.018*
Interaction Dosage/Temperature	< 0,000*	0.070	< 0.000*	0.460

* Significant effect ($p < 0.05$).
Source: Authors.

Table 4: Efficiency of clarification (%) with respect to the physicochemical parameters of conductivity ash, color, turbidity and filterability of the clarified sugar syrup as a function of temperature and the dosage of cationic surfactant.

	Conductivity ash ε **(%)**			**Color ICUMSA** ε **(%)**			**Turbidity** ε **(%)**			**Filterability** ε **(%)**		
Temperature [°C] → / **Surfactant Dosage [ppm] ↓**	**26**	**40**	**55**	**26**	**40**	**55**	**26**	**40**	**55**	**26**	**40**	**55**
150	$22^c \pm 2$	$21^c \pm 2$	$22^c \pm 2$	-28 ±8	-32 ±4	-29 ±6	$-36^c \pm 0$	$-44^c \pm 1$	$-23^c \pm 1$	$64^a \pm 3$	$65^a \pm 1$	$67^a \pm 2$
225	$34^b \pm 1$	$32^b \pm 2$	$28^b \pm 2$	-23 ±6	-36 ±6	-32 ±2	$-77^b \pm 0$	$-98^a \pm 0$	$-98^a \pm 0$	$48^b \pm 2$	$50^b \pm 1$	$51^b \pm 2$
300	$39^a \pm 2$	$39^a \pm 2$	$42^a \pm 2$	-28 ±1	-24 ±6	-25 ±9	$-99^a \pm 0$	$-80^b \pm 1$	$-72^b \pm 0$	$31^c \pm 1$	$30^c \pm 1$	$32^c \pm 1$

± Efficiency preceded by a negative sign (-) indicates a reduction of the parameter compared to the untreated sample, and without any sign indicates that there was an increase of the parameter analyzed.
[a,b]: Efficiency followed by the same letter, vertically, do not differ statistically by the Tukey test, at 5% confidence level.
Source: Authors.

The experimental results of conductivity ash (Fig. 2 and Tables 3 and 4) showed an increase at all temperatures and dosages of cationic surfactant. Albuquerque (2009) observed that higher temperatures could favor the removal of the ash content of the clarified sugar syrup, since heating benefits precipitation of significant amounts of certain compounds responsible for the increase in the ash contents, due to the lower solubility at elevated temperatures. In the temperature range used in this study, such removal was not observed. The lowest results of ash content for the clarified sugar syrups by DAF were observed at 150 ppm dosage in the three temperatures investigated. Statistical analysis for the results of ash content showed significant differences ($p<0.05$) at all temperatures (26, 40 and 55°C) and for all cationic surfactant dosages evaluated (150, 225 and 300 ppm).

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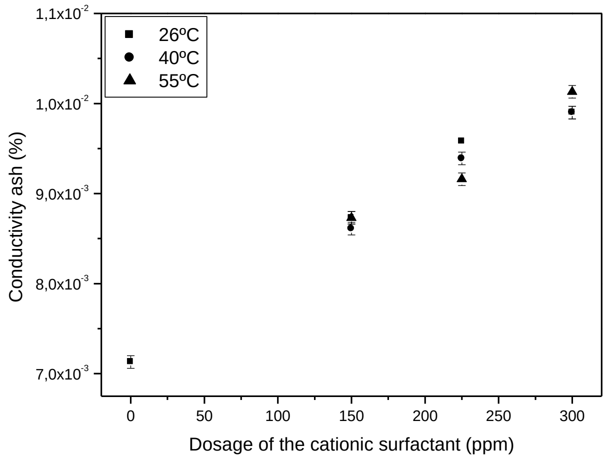

Fig. 2: Conductivity ash of the clarified sugar syrup as a function of temperature and dosage of the cationic surfactant.

Source: Authors.

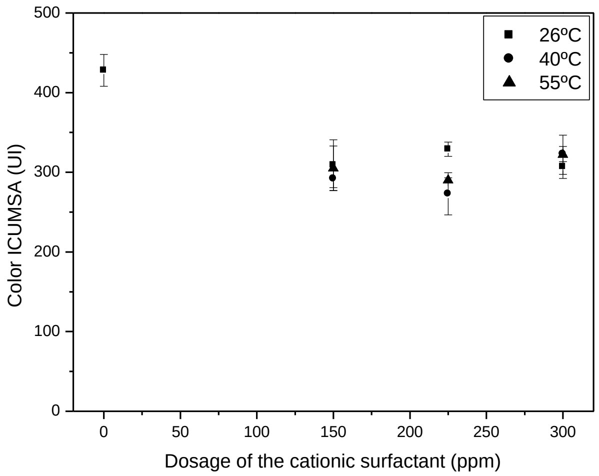

Fig. 3: Color ICUMSA of the clarified sugar syrup as a function of temperature and dosage of the cationic surfactant.

Source: Authors.

It can be noticed at all dosages of the cationic surfactant used in the experiments, that there was color removal (Fig. 3 and Tables 3 and 4) compared to the untreated syrup (0 ppm), at least with 23% color removal (225 ppm, 26°C) and a maximum of 36% color removal (225 ppm, 40°C). The most favorable temperatures for the removal of color were 40 and 55°C. Thus, it can be inferred that the best outcome for the color of the clarified sugar syrup obtained by the DAF process, compared to the untreated samples, was at a dosage of 225 ppm and temperature of 40°C. The temperature and dosage of cationic surfactant showed no significant differences ($p > 0.05$) for the results of color of the clarified sugar syrup by DAF.

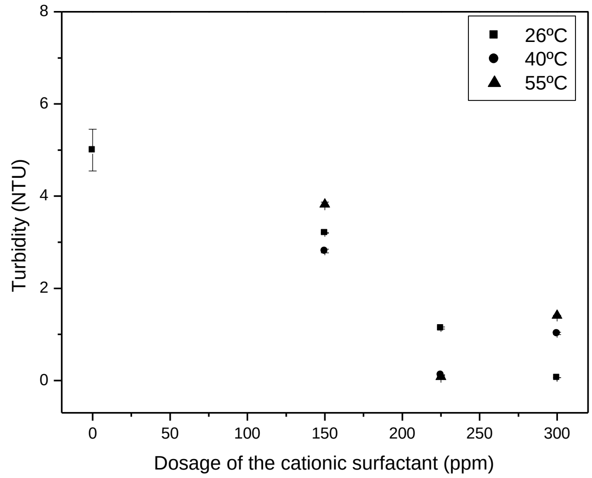

Fig. 4: Turbidity of the clarified sugar syrup as a function of temperature and dosage of the cationic surfactant.

Source: Authors.

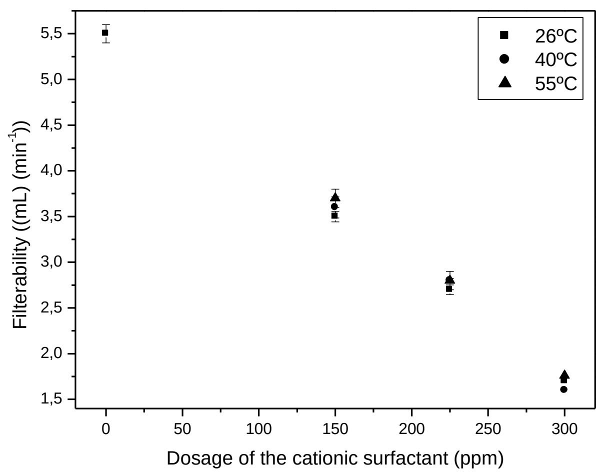

Fig. 5: Filterability of clarified sugar syrup as a function of temperature and dosage of the cationic surfactant.

Source: Authors.

The experimental results for the turbidity of the clarified sugar syrup (Figure 4 and Tables 3 and 4) show that at all temperatures and at dosages of 225 and 300 ppm of cationic surfactant there was a great reduction in turbidity as compared to the untreated syrup. The best results were obtained at 26°C and 300 ppm of the cationic surfactant, and at temperatures of 40 and 55ºC and 225 ppm of the cationic surfactant showing 98% reduction or greater in turbidity of the clarified sugar syrup compared to the untreated syrup. Such combinations of treatments provided clear sugar syrups, indicating the removal of dispersed colloids in the syrup by the DAF process. The range of temperature (26-55ºC) and dosages of cationic surfactant (150-300 ppm) investigated as well as the interaction between them has

significantly influenced positively the turbidity ($p > 0.05$), in a way that higher temperatures implies lower dosage of cationic surfactant to obtain a clear syrup.

Fig. 5 and Tables 3 and 4 show that a higher dosage of the cationic surfactant has negatively influenced on filterability of the clarified sugar syrup compared to the untreated syrup. Note that an increase in the filterability implies in better performance (positive $\varepsilon(\%)$) of the clarified treated syrup, differently from the other physicochemical parameters. This fact could be observed at the lowest dosage (150 ppm) at all temperatures investigated (26, 40 and 55°C), in which a greater volume of filtrate in relation to other dosages was obtained, indicating that there was a significant removal of undesirable high molecular weight substances in the clarified sugar syrup by the DAF process such as starch and dextran. Statistical analysis indicated a significant ($p<0.05$) effect of temperature and dosage of the cationic surfactant on filterability of the clarified sugar syrup.

Although higher dosages of cationic surfactant reduced the rate of filterability, there is a favorable behavior in respect to the temperature, to which we can conclude that the higher the operating temperature the greater the rate of filterability (Figure 5 and Table 3). These results suggest that at higher temperatures, precipitation and removal of unwanted substances present in the syrup is favored, and therefore increasing the DAF process efficiency, even though higher temperatures does not favor the dissolution of air into the liquid medium.

It is interesting to note that the higher the ash content in the clarified sugar syrup the lower the filterability. This fact can be confirmed by the analysis of conductivity ash (Fig. 2), for which the best combination treatment was obtained in the same dosage (150 ppm) and at all temperatures (26, 40 and 55°C) for filterability (Fig. 5). So, the best combination of dosage and temperature, which yielded better results for the filterability of clarified sugar syrup was obtained at 150 ppm of cationic surfactant at temperature of 55°C with an 67% increase in performance for this parameter.

In general, it was observed for the three temperatures (26, 40 and 55°C) and dosages of cationic surfactant (150, 225 and 300 ppm) investigated in this study, 225 ppm of cationic surfactant and 40°C, presented the best results, with the highest removal of color (36%) and turbidity (98%). This same dosage and temperature, the result of filterability rate (50%) presented a satisfactory value, but with an increase in ash content (32%) due to the contribution of the ions present in the cationic surfactant added to the sugar syrup, which were not fully removed by the DAF process.

In study Crema-Cruz et al. (2016) using the same dosages of cationic surfactant at 26°C, applied to another lot of crude sugar crystals (color 355 UI, turbidity 6,00 NTU, conductivity ash 11,0 $.10^{-3}$ % and filterability 6,1 mL min^{-1}) from a different sugar mill, for evaluation of clarification of the sugar syrup by the DAF process, similar color removal, but different results for turbidity and ash from those obtained in the present study, showing the great influence of the raw material used, indicating a need to supplement the DAF process by vacuum filtration or other supplementary technique. According to Spencer and Meade (1967), small differences in total ash content before and after clarification of sugar cane juice may be attributed to changes in the chemical composition and to the addition of auxiliary clarifying agents (eg. calcium hydroxide and phosphoric acid) for the coagulation and precipitation of particles.

Thus, it can be suggested that, for each raw material used in a refinery, chemical treatment given for the coagulation and precipitation of particles to promote the clarification of sugar syrup should be specified according to the quality and composition of the raw material used, and the DAF process may be complemented by filtration to remove ash, in other subsequent stages of production.

IV. CONCLUSIONS

Whichever the temperature, the greater the dose of cationic surfactant, the higher the values of ash contents of the clarified sugar syrup. The dosage of cationic surfactant of 225 ppm at 40°C, yielded better results, with the lightest color (36% removal), highest turbidity removal (98%), and highest rate of filterability (50%), but with an increase in ash content (32%). To help in the removal of ash, the process should be complemented by filtration, or using raw material (crude sugar crystals) with higher quality, facilitating the refinement of crystals and reducing the production cost. The study has demonstrated that it is possible to remove color and turbidity, and to increase in rate of filterability for the clarification of sugar syrup by DAF process by using only the cationic surfactant without adding any other chemical agent, and conducting the process at relatively low temperatures, thus at reduced operational costs.

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